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A device and a method for processing samples

The invention relates to a device and to a method for processing, in particular evaporating samples, according to the preamble of the independent patent claims.

For evaporating dissolved samples held in sample vessels it is known to heat the sample in the vessel. For reducing the boiling point of the solvent it is known to produce a vacuum in the sample vessel. Thus an evaporation at lower temperatures is possible.

Furthermore it is known to move the sample vessel in order to produce swirl in the dissolved sample. The evaporation rate of the solvent is increased by way of this.

It is desirable in an evaporation vessel to be able to process several different samples simultaneously.

Form CH 688 987 there is for example known a reaction chamber into which several sample vessels may be simultaneously placed. The reaction chamber may be placed under a vacuum. The sample vessels are placed in a movable rack which may be driven from the outside via a magnet coupling.

This device however has the disadvantage that complicated mechanisms for driving and heating the sample vessels held in the closed chamber must be provided.

From US 5 569 357 there is likewise known an evaporator with which in the sample solutions a swirl is produced. Also in this device several sample vessels are arranged in a common space.

With all these known devices there exists the problem that the samples in individual sample vessels (which may be of a different nature) may mix via the gaseous phase. The purity of the end product is thus no longer guaranteed.

It is therefore the object of the present invention to avoid the disadvatages of the known, in particular to provide a device and a method for processing, in particular evaporating, which in a simple manner permits the processing, in particular evaporation of several samples held in various sample vessels. The method and the device are to effectively prevent a mixing of the samples via the gaseous phase, are to be simply manufacturable and operable and are to be adaptable in a simple manner to the varying requirements.

According to the invention these objects are achieved with a device and with a method according to the features of the characterising part of the independent patent claims.

The object with the features of the invention serves for processing samples which are contained in sample vessels. The sample vessels comprise at least one filling opening through which the sample may be given into the sample vesssel. In particular the device serves for evaporating the samples.

The device comprises holding means for the simultaneous holding of several sample vessels. The holding means are typically designed as a rack. The usually roughly cylindrical sample vessels (tubelets closed at the bottom) are placed into the rack.

The device furthermore comprises optional heating means for heating the sample contained in the sample vessels. The heating means consist typically of a normal heating plate. With certain

solvents with a low boiling point a heating is not necessary. Evaporation is effected alone on account of the vacuum.

The device is furthermore provided with means for producing a vacuum. Basically all means are suitable which may produce a sufficiently large vacuum. Here and in the following the term vacuum pump is to be understood as all means which may produce such a vacuum.

According to the invention the device comprises connection means by way of which the sample vessels individually or in groups are gas-tightly connectable to a connection of the vacuum pump and by way of this can be evacuated. This design of the connection means comprises several advantages.

Firstly by way of the gas-tight connection of the sample vessels to the vacuum pump a mixture of the samples via the gaseous phase may be prevented. The sample vessels are in principle closed and communicate directly with the vacuum pump.

Secondly the rack and the sample vessels are directly accessible. Since these are not arranged in a closed space, it is possible to directly heat or drive these.

In a preferred embodiment example the device comprises drive means for producing a vortex movement (vortex movement is to be understood as a movement which in the sample vessels produce the desired swirl). The connection means are with this simultaneously designed in a manner such that the holding means and the sample vessels are movable independently of the vacuum pump. In particular the connection means comprise flexible components.

In a further particularly simple embodiment example the connection means comprise at least one connection plate. The connection plate is sealingly pressable onto the filling openings of the sample vessels. Because in the sample vessels there prevails a vacuum there are not necessary any particular provisions for pressing on the connection plate. The pressing plate is sealingly suctioned on. The connection plate is provided with connection paths which serve for connecting the filling openings to the connection of the vacuum pump. So that a sealing connection is guaranteed between the connection plate and the filling openings of the sample vessels there are advantageously arranged sealing means. The sealing means may be exchangeable. Contaminated seals may in this manner be simply exchanged.

The connection plate may in particular comprise longitudinal channels which extend from the lower side, able to face the sample vessels, of the connection plate roughly at right angles to the lower side. The longitudinal channels may be placed on aligned to the filling openings.

The longitudinal channels are connectable via the connection paths to the connection of the vacuum pup. By way of a simple placing of the connection plate onto the filling openings of the sample vessels these are connected to the vacuum pump in a gas-tight manner. A mixing of the samples via the gaseous phase outside the sample vessels is therefore effectively avoided.

In a particularly simple embodiment example the longitudinal channels extend transversely through the connection plate, that is to say up to the upper side distant to the lower side of the connection plate. The connection paths in the connection plate are at the same time formed by deepenings on the upper side. The deepenings communicate with the longitudinal channels. By way of

connection of the longitudinal channels to the connection of the vacuum pump a gas-tight connection between the vacuum pump and the sample vessels may be created. Advantageously with this the level of the exit opening is selected in a manner such that it lies above the level of the deepenings. In this manner there are formed obstacles which prevent a flowing back of condensed fluid into the sample vessels. With this a cross-contamination is avoided.

The connection plate may comprise a connection opening communicating with the collector channels, which is connected or connectable to the connection of the vacuum pump. The connection plate thus serves for connecting the connection of the vacuum pump to the various sample vessels. According to the size and number of the sample vessels there may be provided various holding devices (i.e. racks) and connection plates fitting therewith.

There results a particularly simple design when the collector channels are arranged on the upper side of the connection plate and are open to the top. The sealing results by placing on a sealing plate. Between the sealing plate and the connection plate furthermore along their edge there may be applied a seal.

The sealing plate is preferably manufactured of glass. For avoiding a premature condensation of the solvent on the sealing plate this or also the connection plate may be designed heatable.

The sealing plate and/or the connection plate may furthermore comprise aligning means for centering and firmly holding the connection plate with regard to the holding means.

The above described embodiment example of the connection means is particularly preferred. The connection of the vacuum pump may for example be connected to the connection openings on the connection plate or plates via one or more flexible tubings.

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Of course also other connection means are conceivable. In place of deepenings arranged on the surface of the connection plate bores may be provided in the inside of a connection plate. In this case a sealing plate may be done away with.

Finally it is also conceivable to connect each of the sample vesses directly via a tubing to a vacuum pump.

Advantageously the holding means and/or the connection means are adaptable to a various number and size of sample vessels. In particular the holding means and/or the connection means may be exchanged according to the size and the number of the applied sample vessels.

In the method according to the invention for processing, in particular for evaporating samples held in sample vessels, preferably there is applied a device as previously described. The samples in the sample vessels are advantageously heated and the sample vessels simultaneously moved. The sample vessels are evacuated individually or in groups. With this the pressure outside the sample vessel is not changed, remaining typically at the surrounding pressure.

The invention is described in more detail in embodiment examples and by way of the drawings. There are shown in:

Figure 1 a schematic representation of a device according to the invention,

Figure 2 a schematic representation of an alternative embodiment example with a reduced number of sample vessels,

Figure 3 a perspective representation of the connection means and the holding means in an exploded representation,

Figure 4 a perspective representation of the connection plate placed sealingly onto the sample vessels,

Figure 5 a plan view of one embodiment example of a connection plate,

Figure 6 a cross section through the connection plate according to Figure 5,

Figure 7 a schematic representation of the whole device and

Figure 8 a schematic representation of the drive device.

In Figure 1 there are shown the components of a device 1 for evaporating samples which are essential to the invention. The device 1 consists essentially of a housing 29. In the housing 29 there are provided holding means in the form of a rack 2. Into the rack 2 there may be inserted sample vessels 20. The rack is typically manufactured of aluminium. The sample vessels 20 consist typically of glass tubelets or plastic tubelets closed to the bottom.

The rack 2 is placed onto a heating plate 3 (hidden in Figure 1), by which means the sample located in the sample vessels 20 may be heated. The heating plate may be set into motion via a

motor 22 so that in the sample vessels there is produced a vortex movement of the sample.

For reducing the boiling point of the solvent of the probe, in the inside of the sample vessels 20 there is produced a vacuum.

For this there is provided a connection plate 6 which may be sealingly placed onto the filling opening 21 of the sample vessels 20. In the connection plate 6 (see Figures 3 and 4) there are provided connection channels (longitudinal channels 10, deepenings 11). Via the connection channels and a flexible tubing 7 the inside of the sample vessels 20 are connected or connectable to a vacuum pump 4 not shown in Figure 1. The tubing 7 is firstly led into a condenser where the evaporated solvent is condensed (likewise not shown in Figure 1).

In Figure 1 there is shown an embodiment example in which there are provided two connection plates 6. The rack serves for receiving eight times twelve sample vessels. Each one connection plate is placeable onto 48 sample vessels 20.

The device is furthermore provided with a pivotable cover 25 which may be closed for protection during operation.

Figure 2 shows the device 1 according to Figture 1 with a rack 2 and with a connection plate 6 which is adapted to a smaller number of larger sample vessels. In Figure 2 there are shown six sample vessels 20. The sample vessels 20 are sealingly connected to the vacuum pump via a connection plate 6 and for example via three connection tubings 7.

Otherwise the embodiment example according to Figure 2 corresponds to the example according to Figure 1.

Figure 3 shows in an exploded representation schematically the rack 2 and the connection means consisting of the connection plate 6 and of a sealing plate 8.

The rack 2 is formed of a block of aluminium in which there are provided cylindrical holes for receiving the sample vessels.

Onto the sample vessels 22 applied into the holes 31 there may be sealingly placed the connection plate 6. The connection plate 6 is provided with longitudinal channels 10 which extend from the lower side 14 able to face the sample vessels through the connection plate 6. The arrangement and the number of the longitudinal channels 10 is selected such that these may be placed aligned onto the filling openings 21 of the sample vessels 21.

On the upper side 13 of the connection plate 6 there are arranged collector channels in the form of deepenings 11. The deepenings 11 extend from the longitudinal channels 10 and connect these to one another and to a connection opening 9 represented dashed. The connection opening 9 is connectable to the vacuum pump 4 (see the representation in Figure 7).

Onto the upper side 13 of the connection plate 6 there may be sealingly placed on a sealing plate 8. With this all deepenings 11 are sealed towards the top and form collector channels.

The sealing plate 8 is furthermore provided with openings 15 which are aligned with corresponding openings in the connection plate 6 and which serve for aligning and fastening the sealing plate 8 and the connection plate 6 with respect to the rack 2.

In Figure 4 there is shown the connection plate 6 which is sealingly suctioned onto the filling openings 21 of the sample vessels 20. The sealing plate 8 is placed onto the upper side 13 of the connection plate 6.

The sealing plate 8 is furthermore provided with non-shown heating means, for example heating wires or a steamed-on metal layer, which serve for preventing a condensation of the evaporated solvent on the inner side of the sealing plate.

The connection plate 6 is typically manufactured of layered aluminium. The sealing plate 8 consists of glass.

In Figure 5 there is shown a plan view of the connection plate 6. The longitudinal channels 10 extend from the lower side 14 transversely through the connection plate 6 up to its upper side 13. Deepenings 11 connect the longitudinal channels 10 to one another and to the connection opening 9.

In Figure 6 there is shown a cross section through the sealing plate 6. On the lower side 14 as an extension of the longitudinal channels 10 there are arranged in alignment sealing means 16 which permit the sealing pressing of the sealing plate 6 onto the filling openings 21 of the sample vessels 20.

The sealing means 16 are typically formed out of foam-material-like washer 39 or plates. These may be simply changed after a working procedure which eliminates the contamination danger via the seals. The washer 39 or plates are typically placed on a peg 38 which is provided with a bore and which is screwed into the longitudinal channels 10. The washer 39 or the plates may consist of foamed polyethylene. The longitudinal channels 10 end in an exit opening 34. The level 35 of the exit opening lies above

the level 36 of the base of the deepening 11. Thanks to the obstacle formed in this manner the backflow of condensate is avoided. It would however also be conceivable to provide other forms of obstacles, e.g. a projecting bulge.

In Figure 7 there is schematically shown the whole device 1 which consists of the housing 29, the condenser 17 and of a vacuum pump 4. The connection plate 6 is connected via a tubing 7 to a condenser 17. In the condenser 17 the evaporated solvent is condensed and collected in a collector flask. The condenser 17 is connected to a connection 5 of a vacuum pump 4.

Figure 8 shows schematically the drive of the rack which consists essentially of a motor 22 which via a belt drives an eccentric 24. The eccentric moves the heating plate 3 onto which the rack 2 is placed.

The eccentric 24 is furthermore simply adjustable so that the eccentricity e and thus the degree of eccentric movement, i.e. the size of the produced swirl is settable. Likewise also a balance weight 26 may be provided. The balance weight 26 may be adjustable in order to permit an optimal vibration operation. Adjustability may for example be produced in that the distance of the balance weight to the rotational axis is adjustable, e.g. via a thread.